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Study of Microstructural Effects in the Strength of Alumina Using Controlled Flaws

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Strength characteristics were studied as a function of Vickers indentation load for aluminas with two grain sizes. At low loads the strengths tend to well-defined plateaus, the levels of which bear an inverse relation to grain size. These trends are consistent with a transition from indentation-controlled to microstructure-controlled behavior as flaw size diminishes. The conventional indentation fracture formalism was modified to account for this transition.

THERE is a growing realization that evaluations of fracture properties of polycrystalline ceramics using specimens with large-scale cracks may not provide a true indication of flaw response at the microstructural level. In particular, strength trends for ceramics with microstructures of different characteristic dimension appear to be consistent with a systematic increase in material toughness as the ratio of crack size to grain size increases.[1-8] This "toughening" effect may reflect an intrinsic increase from single-crystal to polycrystal properties as the crack grows from subgrain to multigrain dimensions.[1,2,5] In noncubic materials, however, it is possible to explain the same effect in terms of grain-localized "internal" crack-driving forces.[4,7] To date, attempts to incorporate these two factors into a fracture mechanics formulation have been restricted to largely empirical representations of the toughness function. Accordingly, questions as to the mechanics of the failure process, e.g. whether the critical flaw propagates spontaneously from its initial configuration[4] or undergoes some precursor growth prior to instability,[5] remain a matter of some controversy.

This communication investigates the microstructural influence on the toughness characteristics of a noncubic polycrystalline material using indentation flaws. Indentation flaws confer unique advantages in studies of this kind. First, they are well-defined in the configurational sense, and are thus amenable to detailed fracture mechanics analysis.[9] Second, they can be controlled accurately in their size, via the contact load, thereby allowing the gap between microcrack and macrocrack behavior to be bridged. Previous studies on relatively homogeneous materials, notably silicate glasses,[9,10] have established the validity of simple strength/indentation-load relations over a wide range of flaw sizes ($\approx$2 to 100 μm). Microstructural effects should be readily distinguishable by systematic departures from these idealized relations. Finally, the origin of failure is predetermined, so potential complications resulting from competition between different flaw types are eliminated.

The material chosen for experimental study was high-grade alumina,* obtained as disks 25 mm in diameter and 2 mm thick. The specimens were of two nominal grain sizes, 3 μm (AD999) and 20 μm (heat-treated Vistal). Macroscopic toughness measurements on these specimens (using the double-cantilever-beam method) gave 3.9 MPa·m$^{1/2}$ and 4.6 MPa·m$^{1/2}$, respectively.[11] A Vickers indenter was used to introduce a controlled flaw at the center of each disk, at a preselected load within a working range 1 to 300 N. The indentations were made in air, allowed to sit for $\approx$30 min, and then covered with a drop of silicone oil to minimize further access to moisture in the environment. The disks were broken in biaxial tension (flat on 3-point support), inner support diameter 4 mm and outer support diameter 20 mm, with the indentation on the tension side, and the strengths were evaluated from appropriate flexural formulas.[12] Breaking times were kept below 10 s to avoid significant slow crack growth.[13]

Figure 1 shows the strength data as a function of indentation load, $\sigma_f(P)$, for the two representative grain sizes, D. The error bars are standard deviation limits for an average of 15 "good" breaks per point; specimens in which failures initiated away from the indentation sites were excluded from this data set. The curves through the data are theoretical fits. There is a clear trend for the data to reach a plateau at low loads, at levels corresponding to the "natural" strengths of the two materials (as measured on indentation-free controls). This behavior is consistent with a transition from indentation-controlled to microstructure-controlled behavior.

Consider now how this type of transition may be reconciled with conventional indentation fracture mechanics. For equilibrium radial cracks of characteristic dimension C produced at indentation load P and subsequently subjected to an applied tensile stress σ_a, the stress-intensity expression has the general form[9,14]

$$K=\chi P/C^{3/2}+\psi\sigma_a C^{1/2}=K_c^{eff} \tag{1}$$

where χ and ψ are dimensionless quantities which characterize the level of the residual contact field and the crack geometry, respectively. The sole feature distinguishing the present formalism from that previously used for homogeneous materials is in the interpretation of the "effective" toughness term on the right side of Eq. (1), allowing

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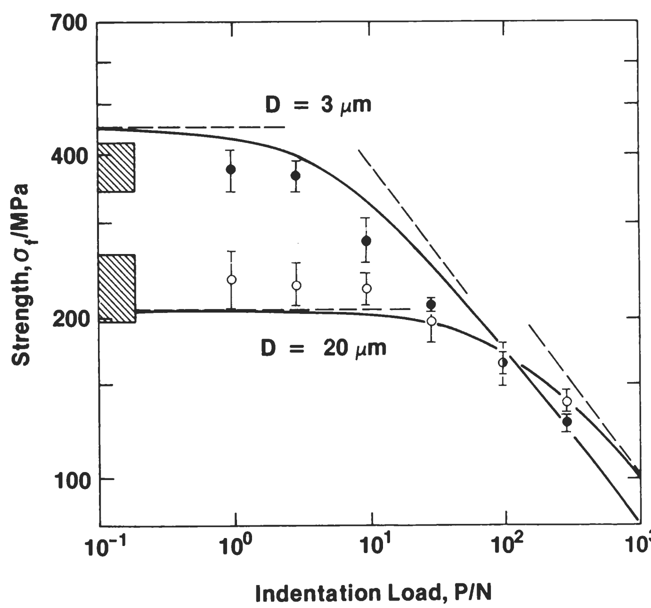

Fig. 1. Inert strength of 3-μm and 20-μm aluminas as a function of Vickers indentation load. Data points represent means and standard deviations for breaks from indentation sites and hatched regions corresponding data for breaks from natural flaws. Solid curves are fits of Eq. (6) to data; broken lines represent asymptotic limits to these fits.

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now for a systematic dependence on the ratio of crack size to grain size, C/D. One convenient way of expressing such a dependence in the region $C/D \geqslant 1$ is[6]

$$K_c^{eff}=K_c^p-\mu K_c^p g(C/D) \tag{2}$$

where K_c^p is the macroscopically determined toughness appropriate to the polycrystalline response and μ is a dimensionless quantity. In this formulation μ may be regarded simply as a numerical coefficient whose origin could lie in either a geometrical (single-crystal to polycrystal) or internal-stress mechanism. The function $g(C/D)$ decreases monotonically, with limiting values $g=1$ at $C/D=1$ and $g=0$ at $C/D=\infty$. Once $g(C/D)$ is specified, Eqs. (1) and (2) may be combined to solve the requisite fracture-stress/load characteristic, $\sigma_f(P)$, in the usual way.[14]

At this point it is useful to consider the stability of the indentation crack system. The presence of the residual contact term in the K formulation of Eq. (1), by virtue of its inverse dependence on C, stabilizes the crack in its initial stages of evolution, producing the very precursor growth characteristic referred to earlier. This characteristic will simply be reinforced by the microstructural element in Eq. (2), since $g(C/D)$ is also inverse in C. Hence, the evolution to failure is not critically sensitive to the exact functional form of $g(C/D)$ (as it is in the case of flaws without residual stress[6,8]). Accordingly, expediency may be taken as grounds for choosing the particular function

$$g(C/D)=(D/C)^{3/2} \tag{3}$$

for which Eqs. (1) and (2) yield explicit solutions. This choice has some physical justification insofar as the microstructural influence may be considered to be grain-localized at the center of the penny-like radial crack, in direct analogy to the geometrical influence of the residual contact zone.[9,15]

The strength/load characteristics are obtained by determining the condition for instability in the function $\sigma_a(C)$ from Eqs. (1) to (3).[14] In its immediate post-indentation configuration, i.e. at $\sigma_a=0$, the crack has dimension

$$C_0=(\chi P/K_c^p+\mu D^{3/2})^{2/3} \tag{4}$$

On application of the external stress, the crack grows stably to a critical size, determined from the condition for a maximum in $\sigma_a(C)$

$$C_m=[4(\chi P/K_c^p+\mu D^{3/2})]^{2/3} \tag{5}$$

Thus the predicted degree of precursor extension for indentation cracks in homogeneous systems, $C_m/C_0 \simeq 2.5$,[9] remains unchanged by the incorporation of the microstructural term. The stress at the critical dimension in Eq. (5) defines the strength,

$$\sigma_f=3K_c^p/[4^{4/3}\psi(\chi P/K_c^p+\mu D^{3/2})^{1/3}] \tag{6}$$

Note that in the limit of large indentation loads this expression reduces to the conventional $P^{-1/3}$ relation for homogeneous materials[9,14]

$$\sigma_f^P=(3/4^{4/3}\psi\chi^{1/3})(K_c^{p\,4/3})/P^{1/3} \tag{7a}$$

In the converse limit of low loads (yet not too low that the cracks are totally encompassed within a single grain), the classical $D^{-1/2}$ relation for grain-limited flaws is obtained[2,16]

$$\sigma_f^D=(3/4^{4/3}\psi\mu^{1/3})K_c^p/D^{1/2} \tag{7b}$$

Hence the transition from indentation-controlled to microstructure-controlled regions emerges naturally from Eq. (6) in terms of upper and lower bounding solutions.

The dimensionless terms in parentheses in Eq. (7) may be regarded as adjustable parameters for data fitting. The solid curves in Fig. 1 were thus generated from Eq. (6) using the values $\psi\chi^{1/3}=2.86$ and $\psi\mu^{1/3}=0.423$ in conjunction with the toughnesses and grain sizes quoted earlier for the two aluminas. It is clear that the theoretical model can account for the general load dependence of the strength behavior. In this context, the relatively pronounced plateau for the coarser grain alumina is worthy of special mention, for it demonstrates that the range of influence of the microstructural term (as delineated, for example, by the crossover point for asymptotic solutions) can indeed be substantial. The model also accounts for the trend to a higher plateau for the finer grain alumina, although in this aspect of the data fit there is evidence of some quantitative discrepancy. Part of this discrepancy could be due to uncertainty in grain sizes (the nominal grain size representing an average over a spectrum of diameters). Part could also be due to the fact that the individual quantities ψ, χ, and μ are not necessarily material-independent, as implicitly assumed in the previous parameter adjustments; sources of variation in ψ and χ are discussed elsewhere,[17] and will be manifest in μ if the underlying microstructural fracture mechanics involves other than geometrically similar processes.

This last point concerning the μ term bears some elaboration, because so far it has been treated as no more than an undetermined coefficient. In this sense the model is phenomenological. As indicated earlier, the origin of the effective toughness variation in Eq. (2) could lie in an increase from single-crystal to polycrystal values, in which case $\mu=1-K_c^s/K_c^p$ could be written, where K_c^s is the critical stress-intensity factor for single crystals[6,8] (consistent with the limiting values of g). Alternatively, it could (in noncubic materials) lie in a concentrated microstress state within a central grain, e.g. resulting from thermal-expansion anisotropy or elastic mismatch (second phase),[7] which gives $\mu \propto \sigma_i D^{1/2}/K_c^p$, where σ_i is the internal stress level.[18] Note that μ is material-dependent in both cases, but in different ways. In principle, it should be possible to determine the relative importance of these contributing factors from the toughness and grain-size dependencies of the strength characteristics, but this determination would require a more extensive investigation of well-defined material systems, with greater control over microstructural properties, than is presented in this work. Systematic studies of single-phase materials as a function of temperature[7] or of materials which undergo cubic-noncubic transformations (e.g. $BaTiO_3$)[19] could also provide definitive evidence of the relative importance of these factors.

Finally, some implications of the results in Fig. 1 concerning design criteria may be considered. It becomes apparent that the macroscopically determined toughness K_c^p is not a reliable parameter for describing strength properties at the microstructural level. The strengths in the plateau region are significantly lower than those obtained by extrapolations from the high-load, indentation-controlled region. Moreover, the fact that the two curves in Fig. 1 can cross over at intermediate loads shows that the macroscopic toughness may not even be a useful *qualitative* indicator of load-bearing capacity. Thus in the present experiments, it is the alumina with the lower value of K_c^p which, by virtue of its finer microstructure, has the superior strength response in the low-load region. This trend to higher strengths with refined microstructures is reflected in the flaw-size relation from Eq. (4) in the limit of small P, $C_0=\mu^{2/3}D$, which dictates the fracture mechanics in the plateau region. The plateau strengths themselves may constitute the most suitable design parameters, not only because they relate directly to crack behavior in the domain of naturally occurring flaws but also because of their insensitivity to extraneous contact-related events, e.g. as might be incurred in machining operations during finishing or in spurious particle impacts during service.

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